DEFORMATION EFFECTS ON DISPERSION HARDENED ALLOYS

by

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ABSTRACT

The investigation of creep and creep-rupture properties in the TD-nickel alloy system is proceeding along lines directed towards controlling the properties by controlling the grain size of the alloy. The preliminary study is nearly complete. Suitable variance of grain size and shape--material variables to be examined in the creep and creep-rupture work--has been established with 3" bar TD-nickel starting material, and efforts are continuing to extend this degree of microstructural control into TD-nickel and TD-nickel-chromium sheet materials.

The capability for elevated temperature tensile testing has been established, and testing is in progress to provide basis data for later creep and creep-rupture investigations. The required experimental apparatus for creep and creep rupture testing is in the last stages of completion. This equipment will allow testing in an inert argon atmosphere at temperatures as high as 2400°F.

A limited amount of work has been conducted with regard to an internal friction investigation designed to provide independent data regarding elevated temperature creep mechanisms. Attempts to produce single crystals of TD-nickel by conventional and non-conventional electron beam floating zone methods have been unsuccessful.

INTRODUCTION

To some degree, the fabrication and use of thoria dispersed nickel base alloys has surpassed a well-grounded understanding of the factors which impart their excellent elevated temperature properties. In an attempt to rectify this situation, work under the present Grant was begun to examine elevated temperature behavior in the TD-nickel alloy system from first principles, and to determine the mechanisms which control it. Such an approach has as its end point an optimization of the elevated temperature performance of these materials.

To date, these investigations have produced significant results. Exploratory examinations of elevated temperature deformation behavior and deformation/annealing responses in TD-nickel bar materials have provided basic guidelines for further, more illuminating, study (1)*. The complex and puzzling deformation and annealing behavior occurring in TD-nickel bar has been well defined and interpreted in terms of the basic controlling variables (2).

These previous studies have established a firm basis for the present phase of the research program (3). This present part of the program has as its goal a determination of the mechanisms controlling the elevated temperature creep and creep-rupture behavior in the TD-nickel alloy system. The present report summarizes research completed during the time period January 1970 to January 1971.

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* Numbers in parentheses indicate reference listings at the end of this report.

INITIAL WORK

Determination of Material Variables:

The material variables considered for potential use in examining creep behavior were: 1) grain size, 2) grain shape, 3) thoria particle dispersion (both volume fraction and mean particle diameter), 4) texturing, and 5) matrix composition.

A complete range of grain size control has now been achieved with TD-nickel 3" bar starting material. Small (0.001 mm average grain diameter), intermediate (0.04 mm diameter), and large (0.5-1.0 mm diameter) grain sizes have been produced using the same starting material (3" bar). Prior to this time, such a range of grain size control has not existed, principally because of the difficulty in producing an intermediate grain size in this material.

Grain size control was developed by following the guidelines established for the deformation and annealing response of TD-nickel bar (2). The small grain size is obtained from the as-received state of the 3" bar (plus a one hour anneal at $2400^{\circ}F$). Abnormal grain growth after approximately 30% cold rolling transverse to the bar axis and annealing for one hour at $2400^{\circ}F$ produces the large grain size. The intermediate grain size is developed by further rolling and annealing of the large grained structure. This involved 75% cold rolling plus a one hour anneal at $2400^{\circ}F$.

The transverse rolling process required to produce a large grain size from the initial fine grain size of the as-received state presented some limitations to the above processing. This was due largely to skin rolling effects which occurred when the initial specimen thickness was greater than 3/8". Skin rolling led to two adverse effects. The first was incomplete (less than 100%) transformation to a large grain size throughout the bulk rolled sample (transformation occurred in preferential areas of the specimen). The second effect was a mechanical splitting fracture of the rolled samples at the middle of their thickness (i.e. an "alligatoring" effect), which occurred at reduction levels near 30%. The tendency for "alligatoring" diminished as the reduction per pass was increased.

To eliminate the above, an initial rolling specimen geometry of 2" in the rolling direction x 1" wide x 3/8" thick was adopted. This specimen (machined from the as-received state of the 3" bar) was then rolled down to ¼" thick in reductions of 0.050" per pass, rotating the specimen 180° about the rolling direction axis and about the normal to this axis after each pass to produce more uniform deformation. After a one hour anneal at 2400°F, this material was 100% transformed to a large grain size. The intermediate grain size was obtained by rolling the annealed ¼" specimens to a 0.075" thickness in reductions of 0.030" per pass and then annealing one hour at 2400°F. No tendency for "alligatoring" was encountered for this second rolling process.

Grain shape control is being achieved by swaging of the as-received state of TD-nickel 3" bar.* The criteria for optimal variation of grain shape are: 1) that the grain size remain constant, 2) that textural variations with variation of grain shape be minimal or at least simple in nature, and 3) that the grain shapes obtained be uniform and relatively simple in geometry.

These criteria are best fulfilled by employing swaging deformations, along with a standard anneal of one hour at 2400°F. Previous work has shown (2) that such a treatment, while altering the grain shape, does not change the grain size. Furthermore, textural changes produced by swaging (the as-received state of TD-nickel 3" bar possesses a weak $\langle 100 \rangle$ - $\langle 111 \rangle$ fiber texture) are not complex, as swaging tends only to strengthen (and slightly alter) the fiber texture already existent. Finally, grain shape changes on swaging are relatively simple in nature. The initial as-received state of 3" bar contains approximately equiaxed grains. With swaging, these become elongated in the swaging direction and their shapes changed to that of a prolate spheroid (an ellipse rotated around its major axis).

In order to produce significantly different changes in grain shape while retaining enough material from which to make reasonably-sized mechanical test specimens, an initial diameter of 1" was required for swaging. The as-received state of 3" bar serves as the

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* NASA facilities at the Lewis Research Center, Cleveland, Ohio were utilized.

equiaxed grain shape. This equiaxed state has a length-to-diameter ratio (or L/D ratio) of 1. An intermediate elongated grain shape is then produced by swaging to a 0.464" diameter (to produce an L/D ratio of 10). Large swaging reductions to a 0.322" diameter are used to obtain a highly elongated grain shape (L/D ratio of 30), which approximates that occurring in TD-nickel commercial 1" bar. All swaging reductions were cold, with no intermediate anneals. Swaging was followed by a one hour anneal at 2400^{o}F.

The various grain sizes and grain shapes produced from 3" bar TD-nickel will be characterized by quantitative microscopy, transmission electron microscopy (to examine substructure), and textural measurements. With regard to textural measurements, the capability of producing complete pole figures is presently at hand. The experimental equipment involves use of the x-ray diffractometer together with a GE Integrating Pole Figure Goniometer. Specimens must be thinned to 0.001-0.002" thick for the application of this method. This is accomplished most satisfactorily by spark machining.

A pole figure obtained for the as-received state of 3" bar TD-nickel is shown in Figure 1. To assemble the pole figures, a randomly oriented sample of fine grained TD-nickel bar consolidate (100% dense) was used as a standard. This material was obtained from Fansteel Corporation (a randomly oriented TD-nickel sheet consolidate was also obtained). For the large grained materials, which are not amenable to the above standard pole figure experimental technique, a suitable pole figure will be constructured from Laue back reflection photographs of individual large grains.

The material variables of thoria particle dispersion and texturing will probably not be examined extensively in the near future. The thoria particle dispersion cannot be varied satisfactorily in the laboratory (e.g. by thoria particle coarsening, for example). Thus, an examination of this variable must rely on material supplied by the commercial supplier of TD-nickel alloys, the Fansteel Corporation. Such material can be supplied by Fansteel, in the form of experimental 10 pound lots of both TD-nickel and TD-nickel-chromium. Such material is in a fine grained, as-forged, consolidated condition and is most easily provided as 3/4" thick plate. A variation of thoria volume fraction from 0 - 10% is possible and the thoria particle size may also be altered. However, a major deterent to the study of the variable of thoria particle dispersion is the high cost* of each experimental 10 pound lot of material. Future budgeting for such a cost may allow the examination of this most interesting and important variable in the future.

To be meaningful, the variable of texturing must be examined independent of any changes in grain size or shape produced as the texture is altered. The Fansteel Corporation can provide TD-nickel and TD-nickel-chromium alloys in a texture-free state. Although a variable texture with constant grain size can be produced (e.g. by the swaging process discussed previously), the production of a variable texture with both constant grain size and grain shape does not seem possible at the present time. Although the potential study of

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*Approximately $1000.00

this variable seems doubtful, familiarity gained with the TD-nickel alloy system might provide some routes for its examination.

The variable of matrix composition will be examined by comparing the behaviors of TD-nickel 3" bar and TD-nickel sheet material to that of TD-nickel-chromium sheet material. TD-nickel-chromium bar cannot be examined in this study because of the prohibitive material cost* and the non-homogeneous as-received properties of the bar material.

A program is presently underway to evaluate the deformation and annealing properties of TD-nickel and TD-nickel-chromium sheet, on the basis of what has been achieved in TD-nickel 3" bar material. The Fansteel Corporation has provided small samples of these materials for this purpose. The goal here is to produce the same degree of control over grain size and shape in the sheet materials, as was possible in the 3" bar TD-nickel alloy. An inherent difficulty in this task may be the greater resistance of the TD-nickel-chromium alloy to mechanical deformation (4). Ideally, grain size and shape control should be produced from the same initial starting state of the material by suitable deformation and annealing treatments. For TD-nickel, indications are that this may be possible if the starting material is plate of ¼" or greater thickness. However, for the TD-nickel-chromium alloy, it may not be possible to achieve such an ideal situation. This would mean that the commercial supplier (Fansteel Corporation) would have to provide fabricated materials of different grain sizes and shapes.

*For 3" bar, the Fansteel Corporation requires a minimum order of 100 pounds, at a cost of $100 per pound.

Elevated Temperature Tensile Testing:

The capability for conducting tensile tests at temperatures from room temperature to the melting point of nickel (2650°F) at a vacuum of 5×10^{-5} torr has been developed. The experimental apparatus utilizes an Instron tensile testing machine integrally constructed with a Brew high-temperature, high-vacuum furnace. The Instron unit possesses a strain rate range of 0.002 - 2 inches/minute cross-head speed, with a maximum practical full scale load of 5000 pounds and a minimum practical full scale load of 20 pounds.

Tensile specimen geometries which conform to the constraints present in the production of variable grain sizes and grain shapes from 3" bar TD-nickel have been designed and are shown in Figures 2 and 3. Similar geometries should be suitable for use with TD-nickel and TD-nickel-chromium sheet materials. The major specimen design criterion was that all elongation should occur within a well defined gage section. This was necessary because strain gages cannot be mounted directly on the test pieces when they are to be tested at elevated temperatures. Hence, the test piece elongation must be measured by the tensile machine cross-head motion. To date, all designs have proven to be viable.

The main intent of the tensile testing program is one of providing elevated temperature mechanical deformation data which may be used as a basis for further creep and creep-rupture testing. Of specific interest are yield strength and ductility.

As of the present time, tensile tests have been conducted at room temperature and at various elevated temperatures of interest above $\frac{1}{2}T_m$ (T_m is the melting point) for the different grain sizes produced from TD-nickel 3" bar. These data are shown in Table 1.

CREEP AND CREEP-RUPTURE

To date, all work on this central aspect of the investigative program has been concerned with assembling the experimental equipment required for the proposed study. The objective has been to produce a capability for creep (constant stress) and creep-rupture (constant load) testing in an inert atmosphere (argon) at temperatures as high as 2400°F, within a load range of 5 - 20,000 pounds.

As of the present time, the assembly of the experimental apparatus is nearly complete. Satec Model LD - 20,000# creep units are the basic components. Furnaces capable of 2100°F and 2400°F specimen temperatures have been obtained. These are to be used with metal (2100°F) and ceramic (2400°F) retort assemblies designed to provide a protective argon atmosphere. Creep strain with time will be measured outside the furnace and retort, with an automatic LVDT (Linear Variable Differential Transformer) system monitoring the motion of the creep train hot stud. Both constant stress and constant load conditions may be achieved on the same creep unit by a relatively simple modification of the lever arm loading assembly (5). Specimen designs are the same as those described for elevated temperature tensile testing (Figues 2 and 3).

INTERNAL FRICTION

Work on the internal friction investigation, which has been limited, has concentrated on producing specimens of suitable geometry for the internal friction apparatus (torsion pendulum). The requirement here is a minimum overall length of 4" and a cross-sectional area of approximately 0.0008 in^2. A wire geometry (circular cross-section) is desirable, with a diameter of 0.020"-0.040". However, specimens of rectangular cross-section are also suitable.

It may be possible to produce various grain sizes and shapes in the wire geometry described above from 3" bar TD-nickel by suitable modifications of the deformation and annealing schemes outlined previously. Swaging would be the ideal deformation method, but, while this technique can easily produce various grain shapes, it may be of limited value in producing a range of grain sizes. Should this be the case, a rectangular cross-sectioned specimen might have to be adopted. Specimen preparation techniques should be easily extendable into TD-nickel and TD-nickel-chromium sheet materials.

The internal friction investigation is intended to be an independent means for the consideration of elevated temperature creep mechanisms in the TD-nickel alloy system. Recently, internal friction work of limited scope has been published (6). These data, which were obtained for the behavior of TD-nickel ½" bar, indicate that: 1) the damping in this material is highly anisotropic with respect to specimen orientation (in relation to the bar axis), and 2) broad damping peaks occur in the temperature range 400°C - 650°C which are probably related to grain boundary relaxation processes. In view of these results, a more comprehensive and complete examination of the effects of grain size, grain shape and matrix composition on the internal friction behavior of the TD-nickel alloy system should provide useful data for the analysis of basic creep mechanisms.

SINGLE CRYSTAL TD-NICKEL PRODUCTION

Attempts were made to produce single crystals of TD-nickel from 3" bar TD-nickel starting material, using an electron beam floating zone apparatus. In the initial attempt, 1/8" diameter rods were melted by the floating zone method, a standard procedure for obtaining single crystals from non-dispersion hardened materials. This procedure was found to be completely unsatisfactory, because of a radical alteration of the thoria particle dispersion by the melting process. The appearance of melted and unmelted regions is shown in Figure 4. Thoria dispersion inhomogeneity was confirmed by scanning electron microscopy, using backscattered electrons for the detection of thorium. Figure 5 shows the thoria particle dispersion in melted and unmelted regions similar to those in Figure 4. An interesting feature of Figure 4 is the apparent grain growth which has occurred in the rod when the thoria particle distribution was reduced by melting. On melting, excess thoria was probably removed to the specimen surfaces, as these acquired a grayish-white caste after melting.

Having established these results, the next approach was to heat the 1/8" rod to as close to the melting point as possible, without actually melting the sample. It was hoped that the combination of a slight amount of thoria particle coarsening and experimental conditions (i.e. steep temperature gradients and motion of the hot zone) would induce selective growth of a very small number of grains to produce at least a bi- or tri-crystal. This, however, did not occur and the material remained fine grained after this treatment.

The final approach was to begin the zone refining process with material deformed such that it would transform to a large grain size on normal annealing. A transverse rolling scheme similar to that described previously for 3" bar material was used. One-eighth inch rods were machined from this rolled material. A nickel seed crystal was then melted onto the specimen to provide a nuclei for grain growth, and the zone refining process begun at a temperature below the melting point. The intent was to aid selective grain growth by: 1) providing an external nuclei, and 2) using material which was inherently prone to large grain growth on high temperature annealing. However, again this process failed to produce a single, bi-, or tri-crystal. Instead, the resultant microstructure was similar to that produced by ordinary annealing.

In view of the previous results, it seems doubtful that a single crystal of TD-nickel can be produced by conventional electron beam floating zone methods, even with extensive modification of technique. A possible alternative approach is the use of internal oxidation to produce a ThO_2 dispersion in an initial nickel-thorium alloy single crystal. However, it is not know whether a suitable thoria dispersion could be achieved by this method.

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TABLE 1 - Tensile Test Results for Various Grain Sizes

Test Temp(^{O}F)	Yield Strength(PSI)	Tensile Strength(PSI)	Elongation (%)
	Small grain size (0.001 mm)		
75	77,800	97,700	7.5
	81,200	102,000	8.5
1100	9,840	10,200	41.8
1500	5,960	5,960	7.2
1800	3,320	3,650	8.85
	3,160	3,320	13.5
2000	1,260	2,120	8.5
	1,470	1,860	13
	1,350	1,860	16.6
	1,835		
2200	1,150	2,070	11.75
	840	1,380	16.7
	Intermediate grain size (0.04 mm)		
75	56,800	89,500	22
	57,100	88,000	25.2
1100	28,400	32,600	4.75
1500	17,200	18,900	1.7
1800	12,850	13,600	1.7
	11,800	12,650	1.3
2000	8,760	9,290	1.6
	9,420	9,550	1.1
2200	6,290	6,760	1.45
	Large grain size (0.75 mm)		
75	50,400	74,400	22
	51,000	76,100	21
	48,700	74,000	20.5
1100	27,800	29,100	1.7
1500	18,200	18,200	0.1*
1800	12,500	12,500	0.2*
	12,400	12,400	0.1*
2000	10,200	10,200	0.1*
	8,380	8,380	0.1*
	7,610	8,200	1.75**
2200	7,830	7,830	0.05*

* Fractured at or before 0.2% elongation offset.
** Specimen broke into three pieces.

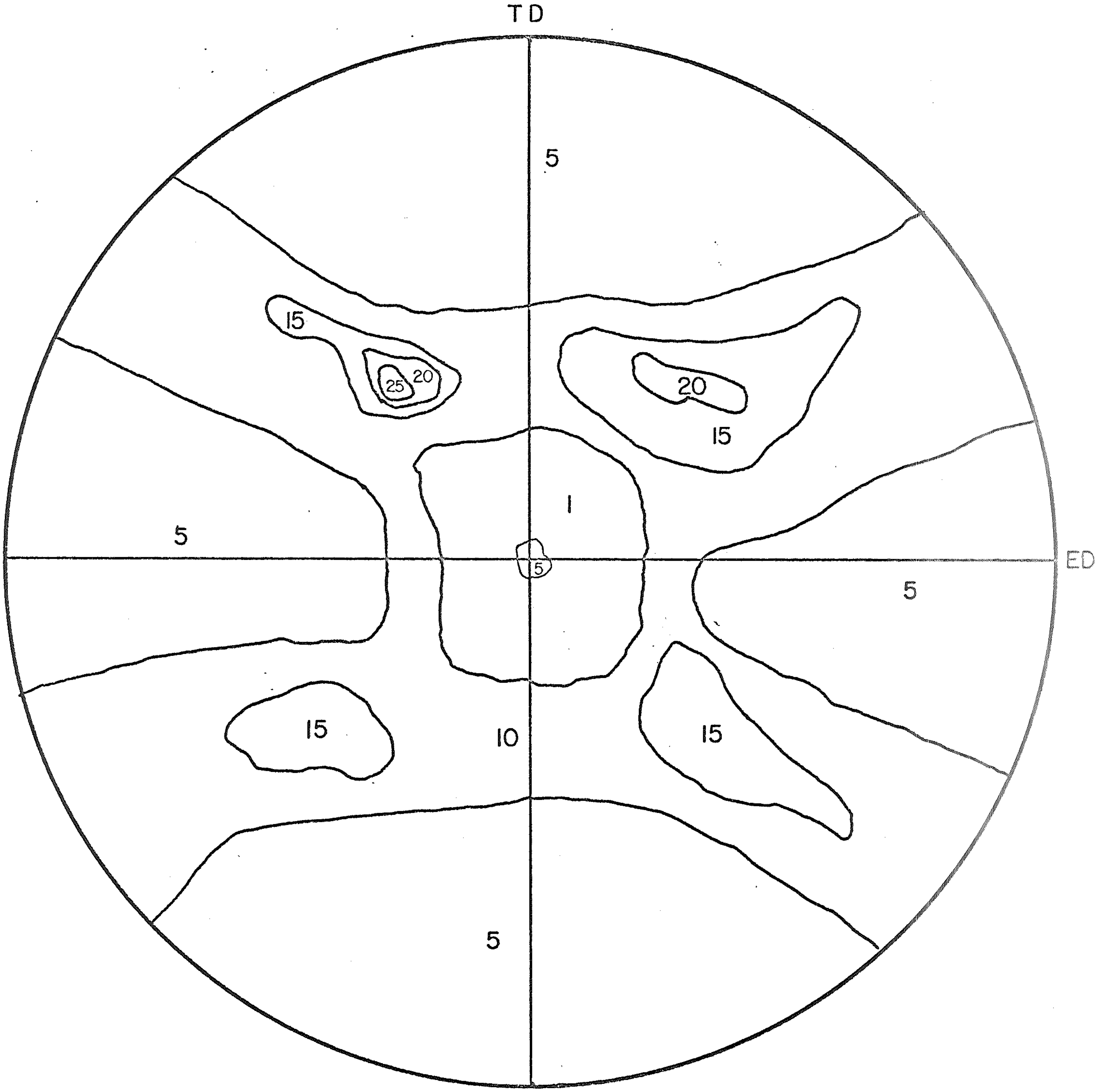

Figure 1 - {111} pole figure of as-received 3" bar TD-nickel. ED and TD refer to the extrusion direction and transverse direction, respectively. Numbers within enclosed regions indicate relative intensities.

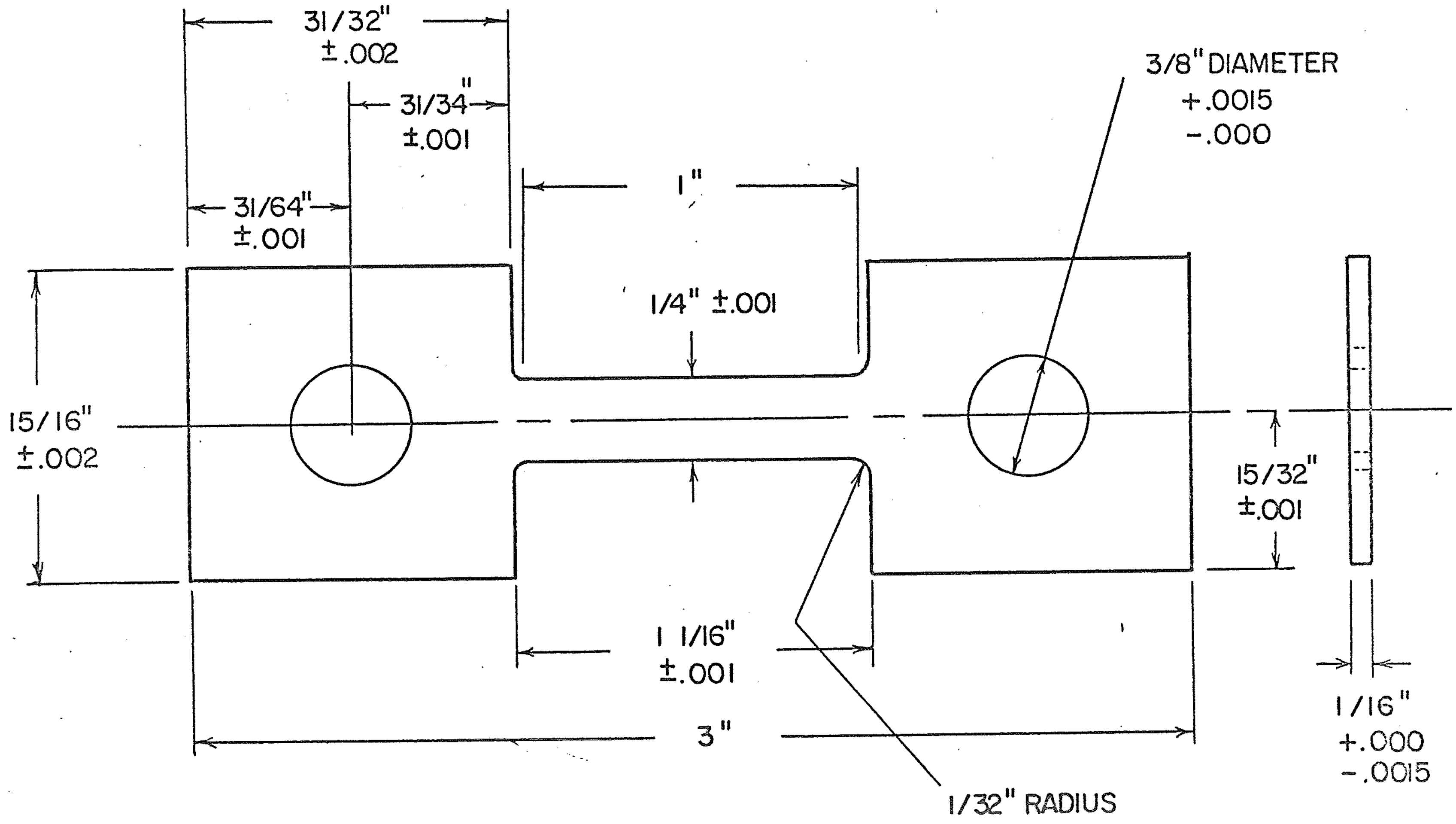

Figure 2 - Sheet specimen for elevated temperature tensile and creep testing.

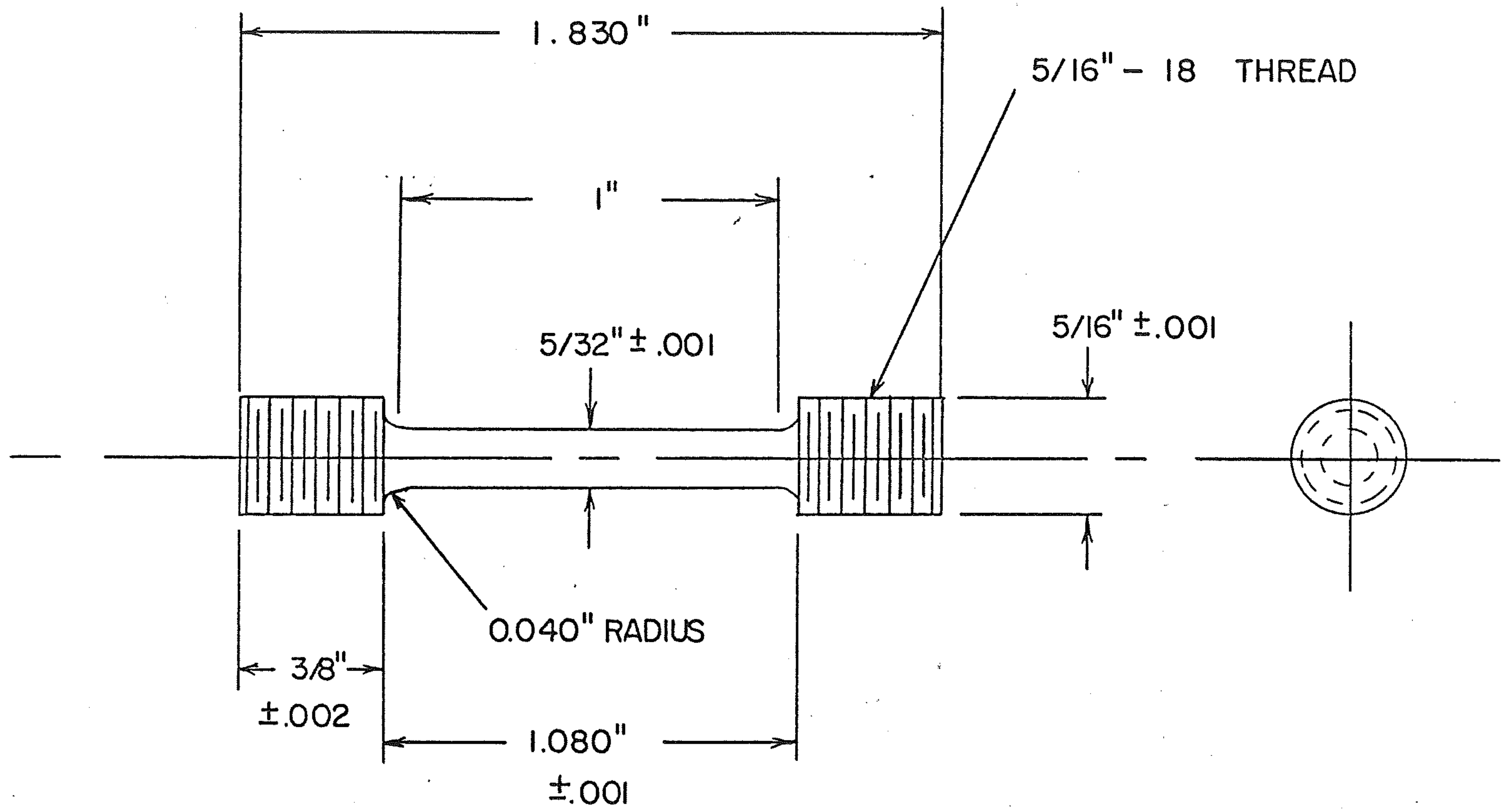

Figure 3 - Round specimen for elevated temperature tensile and creep testing.

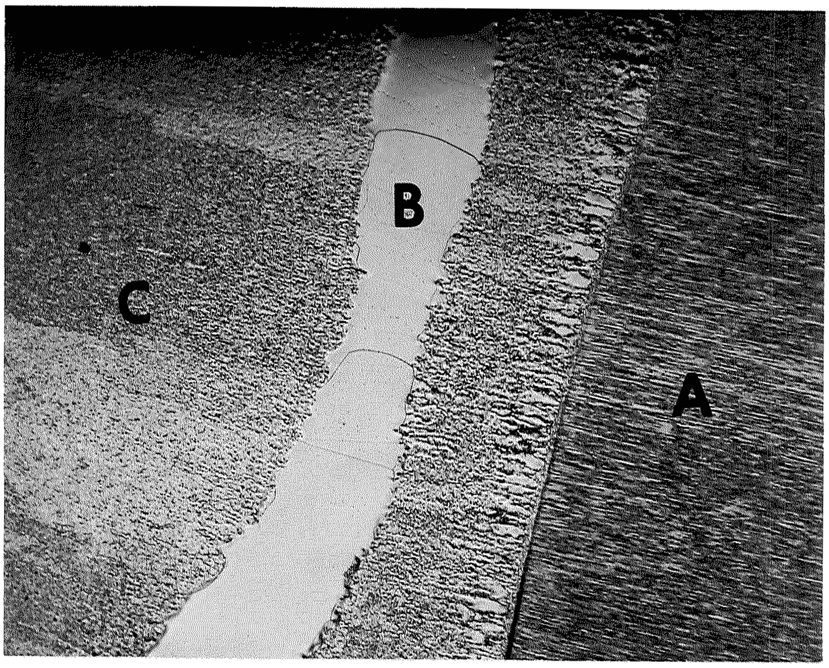

Figure 4 - Appearance of melted and unmelted regions in TD-nickel after electron beam floating zone melting. Unmelted region is at A. Melted region is at B. Note large grain formation at C. (50 x)

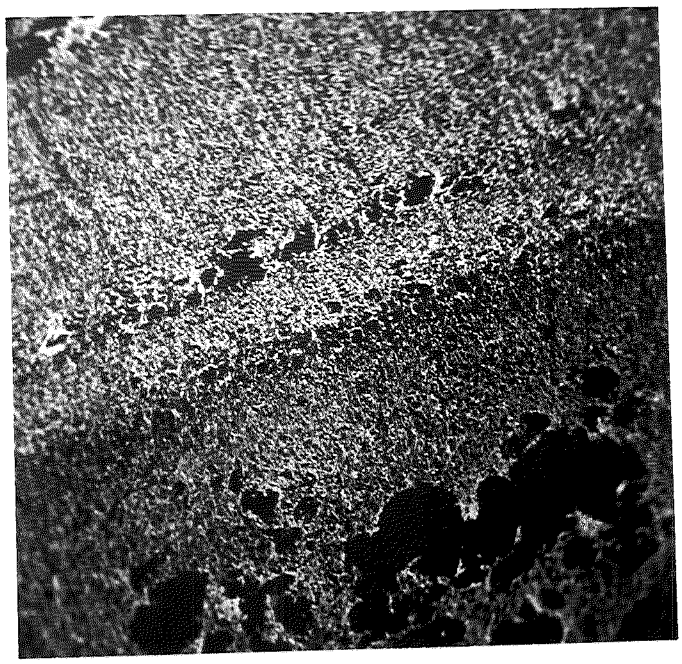

Figure 5 - Scanning electron micrograph of melted and unmelted regions in TD-nickel, using back-scattered electrons. Light regions indicate concentrations of thoria, while dark regions indicate thoria depletion. (200 x)